

All 209 Chlorinated Biphenyl Congeners with an Automated Clean-Up System

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Introduction

The manual sample clean-up for the analysis of all 209 PCBs is described by the US-EPA method 1668C. A combination of a multilayer silica column, florisil column and optional, a carbon column is recommended. The different effect of matrix on the retention of the PCBs on the acidic silica column, depending on their grade of chlorination, has been a challenge transferring the US EPA 1668 method to an automated clean-up system. Some matrices are speeding up the nona- and deca- PCBs, while slowing



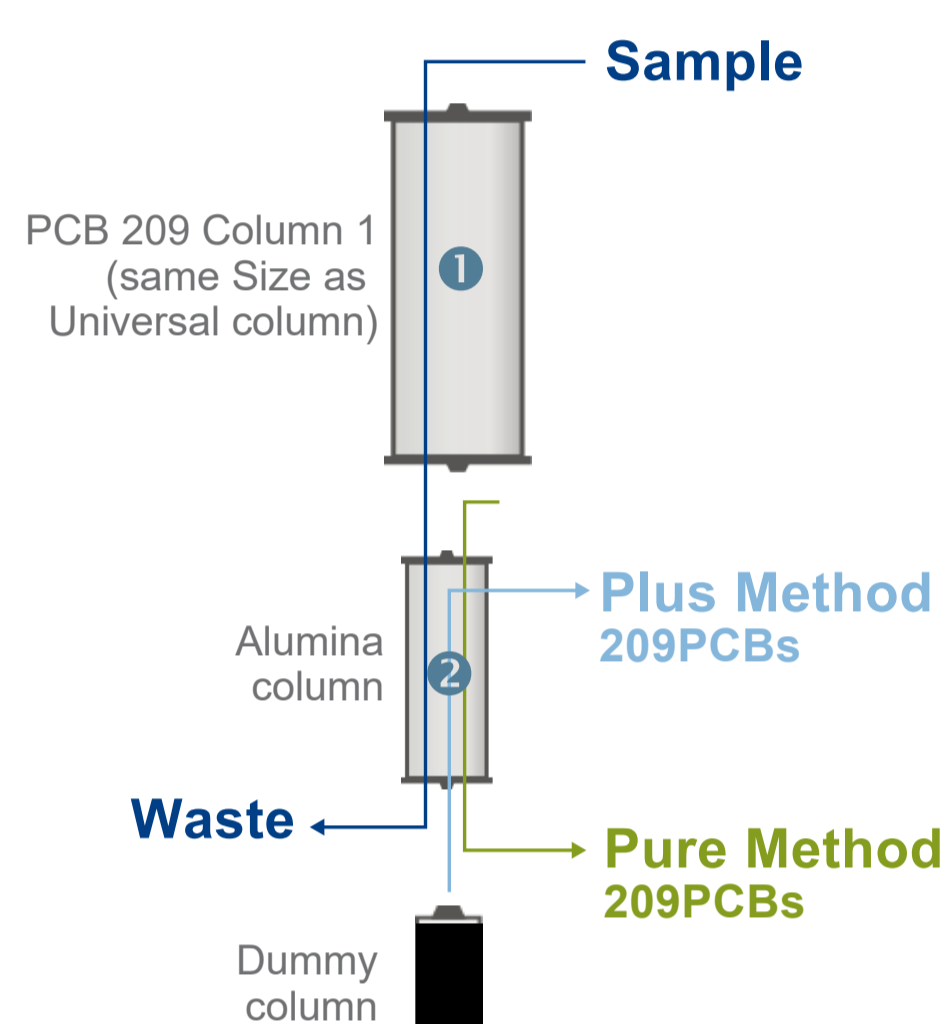
down the mono chlorinated PCBs, which causes the loss of the #209 into the waste or otherwise the elution of interfering compounds into the fraction for analysis and losing mono- and di-PCBs. The automated clean-up method with the DEXTech systems from LCTech minimize

these effects by combining a specially designed acidic silica column with the proven alumina column. Using the rotary evaporator D-EVA from LCTech also enhance the recoveries of the lower chlorinated PCBs!

Material & Methods

1 n-Hexane Sample Loading & Waste

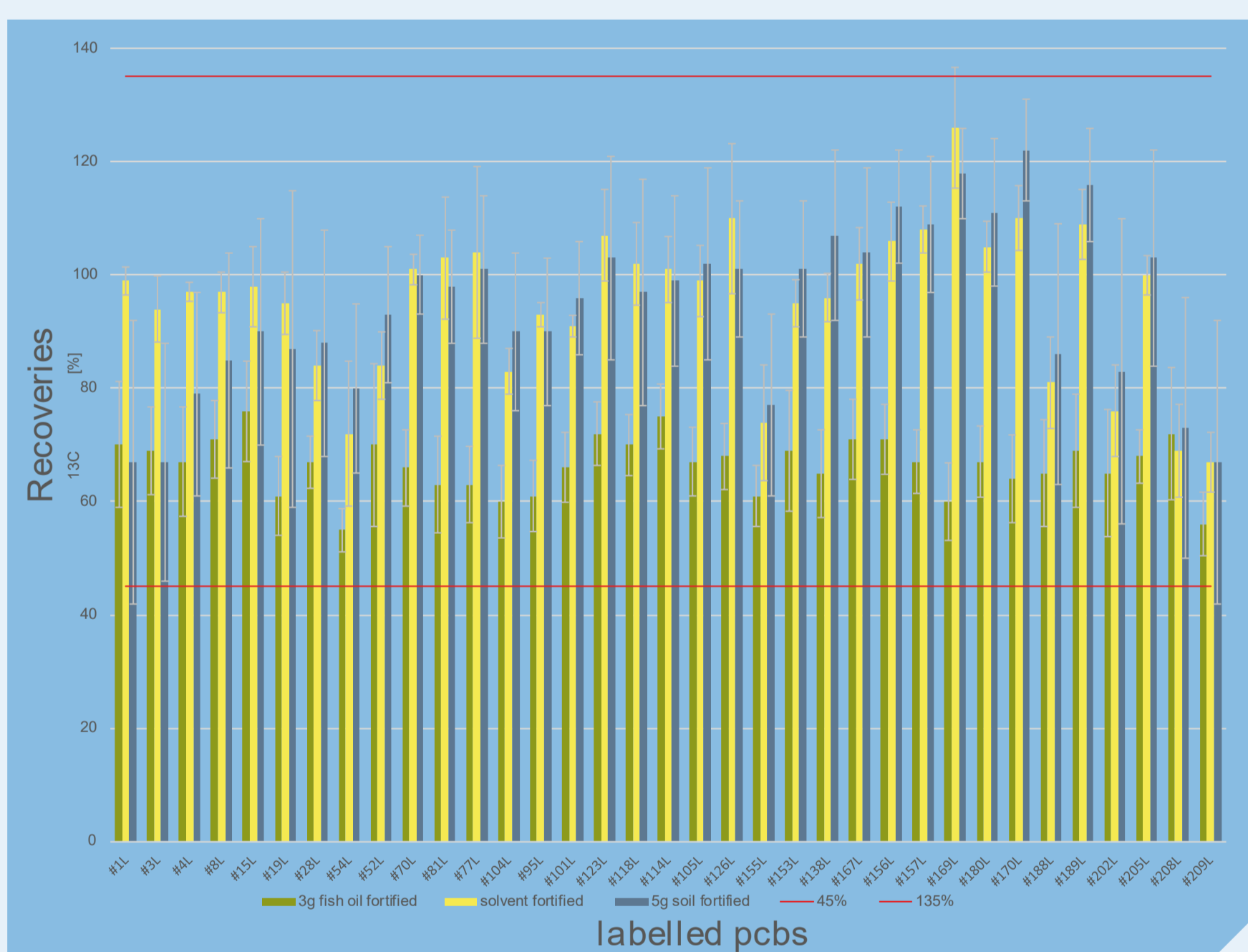
2 DCM / n-Hexan 50/50 Fraction PCB Fraction DCM / n-Hexan 50/50 Fraction PCB Fraction



Besides solvent blanks for the exemplary clean-up of food/feed matrices, 3 g crude fish oil has been directly diluted in n-hexane up to 10 mL total volume. For environmental matrices 5 g dried soil has been extracted with the X-TRACTION® from LCTech, evaporated with the D-EVA from LCTech to 1 mL and filled up to 10 mL with n-hexane. At every test, both samples have been spiked with PCB-LCS-H from Wellington Laboratories directly after the sample intake. For the fortification tests all 209 PCBs have been spiked with AccuGrand-209-PCB from Accu Standards. A DEXTech16 and a DEXTech Pure with a two column setup have

been used for the cleaning alternatively. As first column a specially designed acidic silica column with a size of 150 mm x 25 mm was used. As second column the alumina column from LCTech has been chosen. As solvent n-hexane is used for sample loading on the first column and elution and transfer onto the alumina Column. With dichloromethane/n-hexane 50/50 (v/v) the 209 PCB are collected in a 24 mL fraction. This fraction is evaporated to 100 µL with the D-EVA and spiked with PCB-ISS-H from Wellington Laboratories. The clean extracts are measured with the HRGC-HRMS DFS from Thermo Fisher Scientific, equipped with a SGE HT8 PCB from Trayan.

Results



The diagram in fig.3 shows the recoveries (55-122 %, RSD max 25 %) of the labelled PCB, spiked for the quantification of the fortified matrices and solvent tests. The diagrams in fig 4-6 are showing the recoveries of the native fortification of

- solvent (71-134 %, RSD max 24 %),
- 3 g fish oil (76-128 %, RSD max 13 %)
- extract of 5 g Soil (72-129, max 24 %)

The acceptance criterias of the US EPA method 1668C, table 6, initial precision and recovery (IPR) have been met.

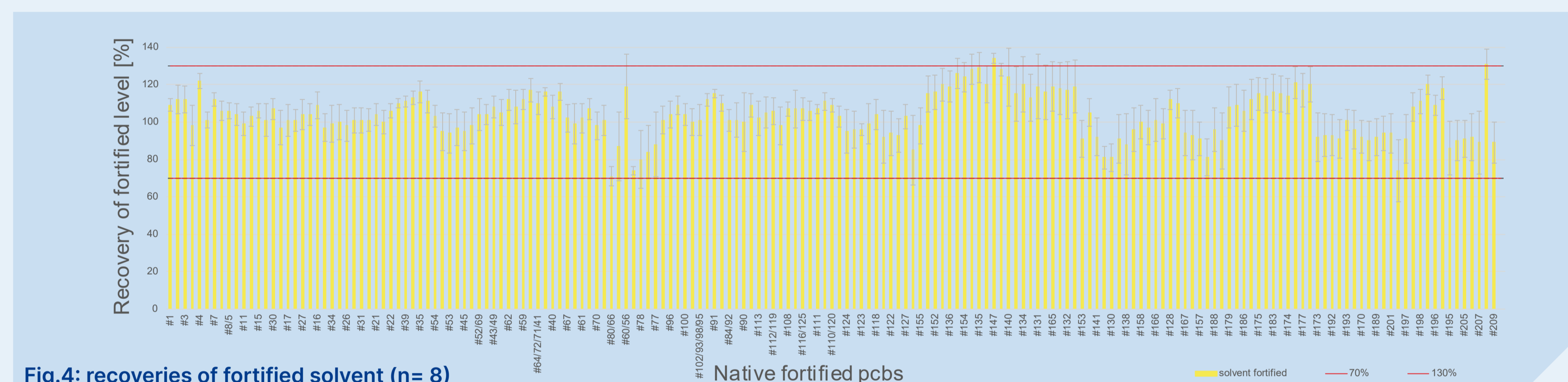


Fig.4: recoveries of fortified solvent (n = 8)

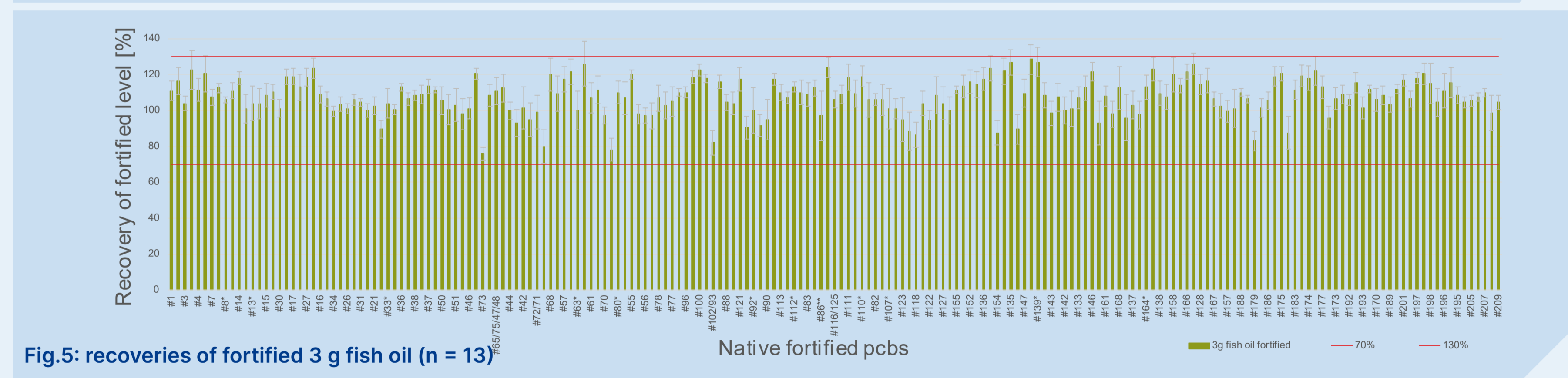


Fig.5: recoveries of fortified 3 g fish oil (n = 13)

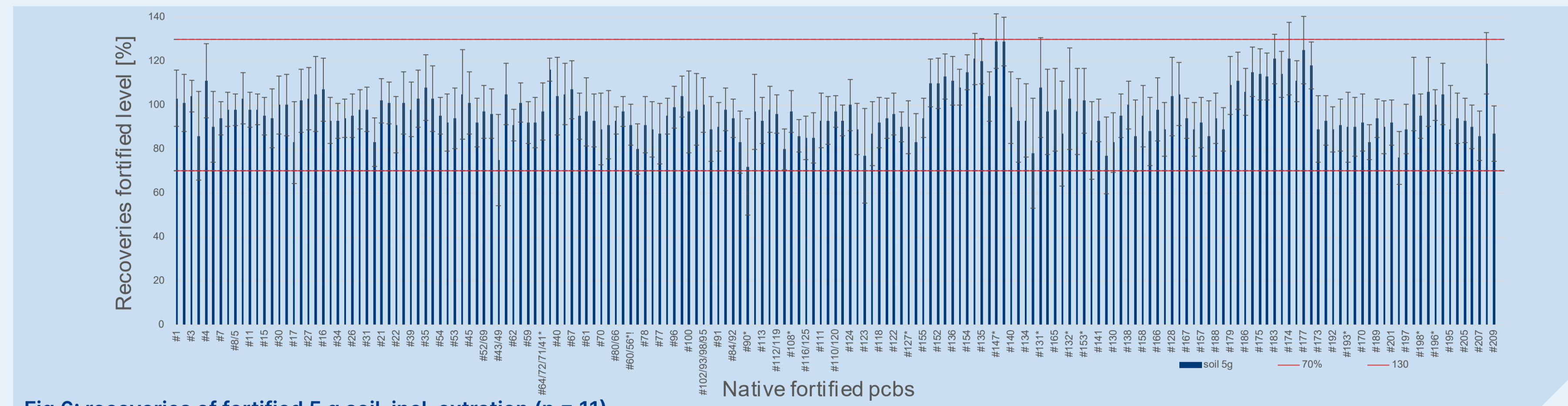


Fig.6: recoveries of fortified 5 g soil, incl. extraction (n = 11)

Conclusion

The here presented results are showing that an automated clean-up of samples for analyzing all 209 PCBs can be done within 33 min, conditioning of fraction lines and columns, quantitative loading of the sample included. The single fraction has a volume of 24 mL dichloromethane/n-hexane 50/50 v/v, that can be easily and fastly evaporated. The total solvent consumption inclusive conditioning and loading is 171 mL with a DEXTech and therefor very economical compared to the US EPA 1668C with

300-350 mL (+50 mL if using the optional Carbon column). The good results of the tests with 5 g soil as matrix, that has to be extracted are showing that the X-TRACTION® from LCTech can be used for the sample preparation as well. Most important conclusion: The advantages of automated clean-up systems (fast, precisely, robust, less solventconsumption ect.) are now available for the PCB 209 method.

2 columns, 1 fraction, 1 measurement!

2 Columns setup	
Process Time (includes loading 4 min and conditioning 8 min)	33 min.
Total Solvent Consumption (incl. conditioning and fractions)	171 mL
1 Fraction	
Fraction Volume (50/50 DCM:n-Hexane)	24 mL
Compared to EPA1668C	
Total Solvent Consumption (incl. conditioning and fractions)	300 - 350 mL

